PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No.:	09/704,881
Filing Date:	November 2, 2000
Applicants:	Richard L. Watkins
Group Art Unit:	1772
Examiner:	Michael C. Miggins
Title:	Process for Improving Interfacial Adhesion in a Laminate
Docket No .:	4022-000007

Director of the United States Patent and Trademark Office
P.O. Box 1450
Alexandria, VA 22313-1450

Appeal Brief Under 37 C.F.R. § 41.37

Sir:

This is an appeal from the Office Action mailed July 6, 2005, finally rejecting claims 1-8, 10-19, and 26-28. A Notice of Appeal was mailed on October 13, 2005 appealing all of the rejected claims. This Appeal Brief is due on December 13, 2005.

This Brief is accompanied by the fee under 37 C.F.R. § 41.20(b)(2).

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Real Party in Interest

The real party in interest is Nike, Inc., a corporation of the State of Oregon, to which the inventors assigned all rights in this invention. The assignment was recorded in the United States Patent and Trademark Office on February 27, 2001 at reel 011543, frame 0796.

Related Appeals and Interferences

An appeal filed in U.S. Patent Application 10,137,531 on October 13, 2005 is related to this appeal. U.S. Patent Application 10,137,531 is a continuation-in-part of the present application. The final rejections of both application rely on Ramesh, U.S. Patent 6,274,228 as a primary reference and cite Wang et al., U.S. Patent No. 6,124,007 and Bonk et al., US 6,082,025 as secondary references.

Status of Claims

Claims 1, 3-8, and 10-29 are pending in the application.

Claims 1-8, 10-19, and 26-28 stand finally rejected.

Claims 20-25 and 29 are objected to as being dependent on a rejected base claim, but would be allowable if rewritten in independent form.

Status of Amendments

No amendment was filed after the final rejection.

Summary of Claimed Subject Matter

Applicants claim a method for improving adhesion between two adjacent layers of a laminate membrane (independent claim 1 and dependent claims 3-17 and 26-28) and a laminate formed according to the method (dependent claims 18 and 19). Claims 20-25 and 29, objected to, are directed to a laminate formed into an inflated bladder and articles including it.

The method of independent claim 1 includes forming a laminate with a first thermoplastic layer adhered to an adjacent second thermoplastic layer and an interfacial boundary between them. There is a lag time during which the laminate is below a temperature at which significant diffusion across the interfacial boundary takes place. Then, the laminate is annealed at a temperature at least about 80°C above a thermal transition temperature of at least one polymeric component of at least one of the layers. The annealing is for a time sufficient for the at least one polymeric component to partially diffuse into the adjacent layer.

"Partially diffuse into the adjacent layer" means that a measurable amount of diffusion takes place, an amount that can be observed by scanning electron microscopy or measured indirectly as an increase in peel strength of the membrane layers. Page 4, lines 1-7. For instance, in comparing Example 1 and Comparative Example A (page 27, line 5 to page 28, line 2 and the table that follows line 2), partial diffusion of a material across the interfacial boundary in Example 1 is evidenced by a four-fold increase in peel adhesion, from 4 pounds per linear inch measured on the non-annealed sample A to 17.6 pound per linear inch measure for annealed sample 1.

The annealing temperature is at least about 80°C above a thermal transition temperature of the diffusing component. A "thermal transition temperature" refers to the

midpoint of a temperature transition region over which the polymeric component exhibits significant changes in properties. Page 6, lines 10-12. The thermal transition temperature may be a glass transition temperature or a crystalline melting point. *Id.* at lines 12-13.

Separately rejected claims

Claim 3 was separately rejected. Claim 3 depends on claim 1 and adds a further limitation of at least one of the first and second layers including a semi-crystalline polymeric component. A semi-crystalline polymeric component has a crystalline melting thermal transition associated with its crystalline regions as well as a glass transition temperature associated with its amorphous regions. Page 9, lines 5-7.

Claim 26, claim 4, and claim 4's dependent claims 16-19, 27, and 28 were separately rejected. Claim 26 depends on claim 1, further requiring at least one polymeric component of at least one of the layers has a glass transition temperature in the range of form about -30°C to 20°C. Polyurethane elastomers prepared with polyester diols, the diols having glass transitions in this range, are described on pages10-11. Claim 4 depends on claim 1, further requiring the first layer to be a thermoplastic elastomer layer and the second layer to be a thermoplastic polymeric barrier layer. These layers are described generally on page 9, lines 1-10. Therrmoplastic elastomers are described in more detail from page 9, line 11 to page 17, line 22; thermoplastic polymeric barrier layers are described in more detail from page 18, lines 1 to 23.

Claims 10-15 were separately rejected. Claim 10 is dependent on claim 4; claims 11-15 are dependent on claim 10. Claim 10 adds a limitation that the laminate is formed into a shape by blow molding before the annealing step. A laminate film of flat or tubular shape may

be blow molded into a desired final shape in a mold having that shape. Page 20, lines 15-23. The blown, shaped laminate is allowed to cool and harden before being removed from the mold. Page 21, lines 4-6. Generally, the annealing should be shortly after blow molded, as set out in claims 11-15, to avoid significant modulus buiding in laminate layers that might interfere with the actions that improve interfacial adhesion during the annealing step. Page 22, lines 3-5.

Grounds of Rejection to Be Reviewed on Appeal

Claims 1, 2, and 5-8 stand rejected under 35 U.S.C. 103(a) as being unpatentable over Ramesh et al., US 6,274,228.

Claim 3 stands rejected under 35 U.S.C. 103(a) as being unpatentable over Ramesh et al., US 6,274,228 in view of Wang et al., U.S. Patent No. 6,124,007.

Claims 4,16-19, and 26-28 stand rejected under 35 U.S.C. 103(a) as being unpatentable over Ramesh et al., US 6,274,228 in view Bonk et al., US 6,082,025.

Claims 10-15 stand rejected under 35 U.S.C. 103(a) as being unpatentable over Ramesh et al., US 6,274,228 in view Bonk et al., US 6,082,025 and Wang et al., U.S. Patent No. 6,124,007.

Argument

I. Claims 1 and 5-8 are patentable over Ramesh et al., US 6,274,228.

The Ramesh patent concerns films that are oriented to be heat-shrinkable. Column 3, lines 43-52. A film is oriented by rapid stretching at a temperature above the glass transition temperature and, for a semi-crystalline material, below the melting point, followed by rapid quenching to preserve the molecular order obtained through the stretching. When the film is heated again to a high enough temperature for molecular movement, the molecules relax and the film shrinks. See, column 6, lines 12-20.

The Ramesh heat shrinkable film has a problem of shrinking along its width at warm storage temperatures. Column 2, lines 13-18. Ramesh teaches that annealing a web of the film at temperatures slightly higher than expected storage temperatures overcomes the problem of width shrinkage, but reduces interlayer bond strength compared to non-annealed films. Column 2, lines 62-67 ("annealed films can exhibit inter-ply bond strength significantly inferior to that of non-annealed films").

In Applicants' method, a laminate is annealed at a temperature at least about 80°C above a thermal transition temperature of at least one polymeric component of at least one of the layers. The annealing is continued for a time sufficient for the at least one polymeric component to partially diffuse into the adjacent layer. Whether at least partial diffusion occurs can be observed indirectly by an increase in peel strength of the membrane layers. Applicants described above the comparison of Example 1 and Comparative Example A, where annealing Example 1 increased the peel strength four fold compared to non-annealed Comparative Example A, demonstrating that partial diffusion occurred.

In comparison, the Ramesh patent teaches that the annealing carried out on the heat shrinkable films *decreases* peel strength compared to non-annealed films. This is not the annealing set out in Applicants' claims; partial diffusion has *not* occurred.

The Ramesh patent sets out to solve the problem of decreased peel strength, while still annealing at the storage temperature to prevent width shrinkage. In the Ramesh modified method, the heat shrinkable film is annealed in a way so that its interlayer adhesion does not decrease as much as in the prior method described in Column 2. The Ramesh patent does *not* teach or suggest that its annealing step increases interlayer adhesion; it merely teaches that the adhesion is not destroyed as it was in the previous method.

Further, Applicants believe it is important to understand that heating an oriented film again to its orientation temperature, a temperature at which molecular movement takes place, causes the film to shrink "almost to its original unstretched, i.e., pre-oriented dimensions." Column 6, lines 18-20. Thus, if the Ramesh annealing step is carried out at a temperature sufficient to allow molecular movement, and for a time sufficient for partial diffusion from one layer into an adjacent layer, the film is going to shrink. It would then be unsuitable for the Ramesh purposes. The Ramesh films are oriented at temperatures between 60 and 140°C; the annealing is carried out at a temperature of greater than 35°C but no more than 100°C, in a way that *preserves* heat shrinkability. Column 14, lines 23-27; column 3, lines 53-56. See also column 27, lines 50-62 (describing necessary free shrink to work effectively in cook-in applications).

Thus, the Ramesh patent does not teach an annealing process for annealing at a temperature above a thermal transition temperature for at time sufficient for at least partial diffusion across the interfacial boundary to take place.

The presently claimed annealing for a time sufficient for partial diffusion does not take place in the Ramesh method. The "annealing" of the Ramesh method may be done at temperatures as low as 35°C and for times as short as a fraction of a second. See column 14, lines 23-53. The temperatures for the Ramesh reheating step are preferably less than about 55°C. See *id.*, lines 39-40. The Ramesh document teaches extremely short exposure times. *Id.*, line 53. The Ramesh film needs to retain its heat-shrink characteristic.

Even were the Ramesh annealing carried out at a higher temperature, the Ramesh reference provides no motivation to maintain that temperature for a time sufficient for the at least one polymeric component to partially diffuse into the adjacent layer. It would appear that molecular movement on that scale is antithetical to the Ramesh film and its required heat-shrinkable characteristic.

The Ramesh patent provides no motivation to alter its conditions or film to attain Applicant's method of intentional diffusion across the interfacial boundary.

Accordingly, and for all of these reasons, Applicants respectfully ask this Honorable Board to REVERSE the final rejection of these claims over the Ramesh references.

II. Claim 3 is patentable over Ramesh et al., US 6,274,228 in view of Wang et al., U.S. Patent No. 6,124,007.

Claim 3 is patentable over the Ramesh patent for the reasons already discussed. The Wang patent does not provide the teachings absent in the Ramesh patent. The Wang angioplasty balloon has two layers of separately-oriented thermoplastic materials. One tube is stretched, inserted into the other, then the other tube is stretched. A post-blowing annealing shrinks the balloon so that it has a "stepped compliance curve" as in Figure 7. The oriented material is partially relaxed. See column 6, line 50 to column 7, line 3. There is no teaching or suggestion of annealing the Wang angioplasty balloon under the conditions of claim 3.

There is, moreover, no motivation to turn to the Wang reference, save in hindsight of Applicant's invention. The Ramesh patent does not appear to require improved flexibility or burst strength. The Wang patent is not concerned with conditions for adhesion between film layers and does not teach selecting materials and conditions that result in inter-laminar diffusion and increased adhesion.. Declaration of Richard L. Watkins. The mere existence of the Wang materials is not sufficient to motivate one to modify the Ramesh method with them.

Accordingly, and for all of these reasons, Applicants respectfully ask this Honorable Board to REVERSE the final rejection of claim 3.

III. Claims 4, 16-19, and 26-28 are patentable over Ramesh et al., US 6,274,228 in view of Bonk et al., US 6,082,025.

These claims are patentable over the Ramesh patent for the reasons given with regard to independent claim 1..

The Bonk reference does not remedy the shortcomings of the Ramesh patent. The Bonk patent does not describe any post-forming heating of its laminate membranes. Further, there is no motivation to turn to the Bonk patent materials to modify the Ramesh patent cook-in laminates. The mere existence of other materials used in laminates in other applications provides no motivation to select them for the Ramesh application or process. Even were one to select other materials, the Ramesh patent does not teach one how to improve interlayer adhesion over an initial adhesion of a laminate by causing a polymeric component to partially diffuse into an adjacent layer.

Accordingly, Applicants respectfully ask this Honorable Board to REVERSE the final rejection of claims 4, 16-19, and 26-28.

IV. Claims 10-15 are patentable over Ramesh et al., US 6,274,228 in view of Bonk et al., US 6,082,025 and Wang et al., U.S. Patent No. 6,124,007.

These claims are patentable over the Ramesh and Bonk patents for the reasons given above with regard to claim 4. The Wang patent forms its angioplasty balloon by stretching two tubes to orient the materials, inserting one tube in the other (before or after stretching the second tube), and pressurizing the tubes at a temperature above ambient. Column 2, line 55 to column 3, line 33.

A post-blowing annealing shrinks the balloon so that it has a "stepped compliance curve" as in Figure 7. The annealing step partially relaxes the oriented material. See column 6, line 52 to column 7, line 3. The Wang patent states that its balloons can be easily peeled apart. Column 6, lines 28-29. The Wang patent does not speculate on whether annealing with its concomitant shrinkage of layers which had been formed independently at different stretch ratios. Applicant's opinion is that one would see no change in interlaminar adhesion. Declaration of Richard L. Watkins, page 2.

Accordingly, Applicants respectfully ask this Honorable Board to REVERSE the final rejection of claims 10-15.

Conclusion

The present claims are patentable over the cited art. Applicants, therefore, respectfully petition this Honorable Board to reverse the final rejection of the claims on each ground and to indicate that all claims are allowable.

Respectfully submitted,

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October 20, 2005

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Claim Appendix

Copy of the Claims Appealed

1. A method for improving adhesion between two adjacent layers of a laminate membrane, comprising the steps of:

(a) forming a laminate having a first thermoplastic layer adhered to an adjacent second thermoplastic layer and having an interfacial boundary between the first thermoplastic layer and the second thermoplastic layer;

(b) after a lag time when the laminate is below a temperature at which significant diffusion across the interfacial boundary takes place, annealing the laminate at a temperature at least about 80°C above a thermal transition temperature of at least one polymeric component of at least one of the layers for a time sufficient for the at least one polymeric component to partially diffuse into the adjacent layer.

3. A method according to claim 1, wherein at least one of the first and second layers includes a semicrystalline polymeric component.

4. A method according to claim 1, wherein the first layer is a thermoplastic elastomer layer and the second layer is a thermoplastic polymeric barrier layer.

5. A method according to claim 1, wherein the laminate is annealed for at least about 15 minutes.

6. A method according to claim 1, wherein the laminate is annealed for at least about 30 minutes.

7. A method according to claim 1, wherein the laminate is annealed for at least about 40 minutes.

8. A method according to claim 1, wherein the laminate membrane is annealed at a temperature above a thermal transition temperature of at least one component of each of the first and second layers.

10. A method according to claim 4, wherein the laminate is formed into a shape by blow molding before the annealing step.

11. A method according to claim 10, wherein the annealing step is carried out within about 2 hours of the blow molding.

12. A method according to claim 10, wherein the annealing step is carried out within about 1.5 hours of the blow molding.

13. A method according to claim 10, wherein the annealing step is carried out within about 1 hour of the blow molding.

14. A method according to claim 10, wherein the annealing step is carried out within about 30 minutes of the blow molding.

15. A method according to claim 10, wherein the annealing step is carried out within about 15 minutes of the blow molding.

16. A method according to claim 4, wherein the annealing step is carried out at a temperature of at least about 100°C.

17. A method according to claim 4, wherein the annealing step is carried out at a temperature of up to about 150°C.

18. A laminate formed according to the method of claim 4, wherein the first layer comprises a thermoplastic polyurethane prepared from a polyester diol and the second layer comprises an ethylene-vinyl alcohol copolymer.

19. A laminate formed according to the method of claim 18, further comprising at least a third layer comprising a thermoplastic polyurethane prepared from a polyester diol that is adjacent to the second layer.

26. A method according to claim 1, wherein at least one of the polymeric components of at least one of the first and second layers has a glass transition temperature in the range of from about -30°C to about 20°C.

27. A method according to claim 4, wherein the thermoplastic elastomer layer comprises a material selected from the group consisting of polyurethanes prepared using polyester, polyether, and polycarbonate diols, flexible polyolefins, styrenic thermoplastic elastomers, polyamide elastomers, polyamide-ether elastomers, polymeric ester-ether elastomers, flexible ionomers, thermoplastic vulcanizates, vulcanized EPDM in polypropylene;, flexible poly(vinyl chloride) homopolymers and copolymers, flexible acrylic polymers, and combinations thereof.

28. A method according to claim 4, wherein the thermoplastic polymeric barrier layer comprises a material selected from the group consisting of ethylene-vinyl alcohol copolymers, vinylidene chloride polymer, acrylonitrile polymer, copolymers of acrylonitrile and methyl acrylate, semicrystalline polyesters, polyethylene terephthalate, polyamides, crystalline polymers, epoxy resins based on N,N-dimethylethylenediamine and resorcinol, polyurethane engineering thermoplastics, and combinations thereof.

EVIDENCE APPENDIX

<u>Evidence entered by examiner and relied on by appellant</u>

Declaration of Richard L. Watkins, entered in the record with the Reply filed June 27, 2002.

<u>Evidence relied on by examiner as to grounds of rejection</u>
Ramesh et al., US 6,274,228
Wang et al., US 6,124,007
Bonk et al., US 6,082,025

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DECLARATION OF RICHARD L. WATKINS UNDER 37 C.F.R. § 1.132

I, Richard L. Watkins, do hereby say and declare that:

1. I am the inventor of the above-identified U.S. patent application.

2. I have been working in the area of laminate membranes for five years.

3. I have read Wang et al., U.S. Patent No. 6,124,007.

In my opinion, the purpose of the annealing step described in the last paragraph of column 6 is to volumetrically shrink the more elastic, outside balloon to provide mechanical compression on the high modulus, inside balloon. The outside balloon shrinks because it has been oriented.

The Wang patent is concerned with strength of the balloons to withstand bursting and is not concerned with interlaminar adhesion. The Wang patent itself

states in column 6 that the balloons can be easily peeled apart. The Wang patent does not claim to improve peel strength at the interface between the two balloons and certainly does not disclose how to improve peel strength. If a person were to select a combination of balloons of any of the materials that Wang describes, and then place one balloon inside the other and subjected the balloons to Wang's annealing conditions for shrinking the elastic layer, I would expect that typically there would be no change in interlaminar adhesion, and any improvement in interlaminar adhesion would be truly accidental.

My invention achieves significant improvements in interfacial adhesion between the laminate layers by annealing at a temperature that is above a thermal transition temperature of one of the layer materials long enough for that material to diffuse into the other layer. The annealing temperature needed for this diffusion to take place changes when different materials are used in the laminate layers. Thermal transition temperatures of materials depend upon many different factors, including the monomer composition of the polymer, the polymer molecular weight distribution, and the chain ends of the polymer. For the specific materials I selected for my working example on page 27 of my patent application specification, annealing at 140°C for twenty minutes was appropriate. In my opinion, the Wang patent does not provide any specific combination of materials and annealing conditions that would lead to the interlayer diffusion of my invention. Wang also does not provide any guidelines for selecting materials and conditions that could result in inter-diffusion and better interfacial adhesion.

4. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true. I understand that willful false statements and the like if made herein would be punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and may jeopardize the validity of the application or any patent issuing therefrom.

Richard L. Watkins

Richard L. Watkins

20-Jun-02

Date

RELATED PROCEEDINGS APPENDIX

There is as yet no decision on the appeal filed in U.S. Patent Application 10,137,531.